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Extraction of Vitamin A From Dogfish Livers 1/

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The world war and the national defense program have created an increased demand for Vitamin A. This vitamin, because of its particular nutritional and therapeutic properties, is an especially vital supplemental factor in the diet of both civilian and military population. The urgency for domestic sources of Vitamin A has been further heightened by disruption of former channels of vitamin trade, and considerable interest is being shown as to the extent of domestic supply and the adequacy of Vitamin A resources to meet the requirements of the nation, both for consumption within its borders and in giving assistance to other countries.

The demand for Vitamin A has led to pronounced stimulation in the utilization of dogfish livers, particularly on the Pacific Coast. Because of this fact, and the evident necessity for maximum vitamin recovery from existing resources, the Fish and Wildlife Service has initiated a study of the efficiency of various methods for extracting Vitamin A applicable to this type of liver material. The accompanying data have been obtained during a cursory survey of the problem, and while they must be considered as being only preliminary in nature it is believed that a sufficient number of points of interest have developed to warrant a report to the industry for their further consideration.

Procedure

The information assembled in the accompanying table represents the results of seven series of experiments on different lots of dogfish livers obtained from local fishing vessels landing market fish in Seattle, during the period April to June. The composition of the raw material, given in the first column of the table, was determined from a representative sample of each lot of livers, the entire lot having been ground and thoroughly mixed before the sample was taken. The individual test lots within each series were made on aliquots (not less than two pounds each) of the entire lot, after grinding and thorough mixing.

The general experimental procedure was based on pilot plant technique, involving semi-commercial grinding, cooking and centrifugal equipment. In the case of series 5, 6 and 7, the cold separation was made by centrifuging the entire lot of ground raw liver material in an imperforate basket centrifuge. The residual liver pulp was then thoroughly mixed and divided into equal portions for subsequent processing, sufficient raw material having been used initially to permit at least two-pound test samples of the residue. All cooks were made with live steam, the cooking time being of ten minutes duration. The percentages of alkali (sodium hydroxide) or acid (sulfuric acid) shown under "Method" are based on the weight of the liver charge, and where the term "alkali then acid," or vice versa, is used, it is meant that the mixture obtained by the alkali or acid cook was partially or totally neutralized, or carried past the neutral point before being centrifuged.

The oil content of the livers (Column 1) was determined by cold ether extraction of equal portions of a composite sample of ground liver and anhydrous sodium sulfate. Vitamin A was determined by the antimony trichloride reaction, absorption of the blue color at 610 mu. being measured by photoelectric colorimeter. The conversion factor for calculating to International units was based on comparable data for a number of the oil samples assayed with a Helger ultraviolet spectrophotometer, in which case a factor of 1850 for E(1%, 1 cm) (328 mu.) was used. Excellent correlation between E(1%, 1 cm) (328 mu.) and E(1%, 1 cm) (610 mu.) was obtained, thus obviating any question of the vitamin data being influenced by abnormalities in the color reaction.

Results

Generally speaking, livers of high oil content, such as those obtained from cod, shark and dogfish, are assumed to respond readily to vitamin and oil extraction. The preliminary results obtained in the present study, however, raise some doubt in this belief. When extractions were made on fresh livers, efficient oil recovery did not necessarily give efficient Vitamin A extraction. Alkali cooks gave oil recoveries approximating 80% to 90% of the total, yet Vitamin A extraction was but 28% to 35% efficient. Water cooks gave equal oil recoveries and about 50% to 60% extraction of Vitamin A. Processing of the fresh liver in a mild acid medium, on the other hand, facilitated Vitamin A extraction, leading to recovery of approximately 80% of the vitamin in about 90% of the oil.

Freezing apparently leads to a physical change in the liver tissue, facilitating Vitamin A extraction. With frozen livers alkali, water and acid cooks did not give wide variation in effectiveness. The results obtained by the three processing methods were similar with respect to both oil and vitamin recovery. The efficiencies of oil and vitamin extraction were closely comparable and relatively high, i.e., 80% to 95% of the oil extracting and removing 78% to 89% of the vitamin.

Brocklesby and his associates at the Fisheries Experiment Station, Prince Rupert, B. C., have demonstrated that adsorption of Vitamin A by the formed soaps incident to alkali processing may lead to low vitamin recoveries. This condition may account to some extent for the poor results obtained in the alkali cooks on fresh liver since considerably greater emulsion difficulty was encountered with the fresh material. The frozen liver experiments suggest also that change can be effected within the liver, influencing the availability of the vitamin for extraction. Thus, the low results on fresh livers are no doubt due to a number of factors. This question is being subjected to investigation.

During the course of the experimental work it was observed that oil breaking naturally from the livers was of considerably lower Vitamin A potency than that of the composite samples. This suggested the possibility of a degree of flexibility in processing high-oil-content livers, leading to removal of part of the oil without appreciable vitamin extraction and recovery of the vitamin remaining in the liver residue in more concentrated form.

Economic Aspects

Dogfish livers are utilized primarily in fortifying poultry oils with Vitamin A and in the preparation of Vitamin A concentrates. In the latter case, oils of higher potency are preferable since the volume of material required in the preparation of a given amount of concentrate will be less and a higher degree of concentration may be possible. For this and other reasons the price paid per million units of Vitamin A in dogfish oil increases with the potency of the oil; thus a gallon of one lot having, for example, four times the vitamin potency of a gallon of another lot of oil may have much more than four times the monetary value.

Ability to concentrate the vitamin in a smaller volume of the oil in the livers would also be of advantage when the vitamin potency of the total oil runs below the value normally acceptable for concentration. The raw or bled oil of lowered potency need not be of definite loss because such oils may still be fortified with Vitamin D, natural or synthetic, for sale in animal feeding.

The poorer availability of Vitamin A in fresh livers makes this material preferable to frozen livers for concentration of the vitamin. The degree of mechanical disintegration influences the rapidity with which the low-vitamin oil separates out, and this in turn influences the degree of vitamin extraction by the raw or bled oil. Coarsely ground livers required several days' aging for the oil to break in quantity.

Cold centrifuging removed 75% and 73% of the total oil present in the livers carrying but 34% and 31% of the total vitamin, respectively. Such a period of aging, however, leads to unfavorable changes in the quality of the liver and oil and cannot be recommended. Fine disintegration of the fresh liver, on the other hand, permits immediate separation of the raw oil in greater quantity and with less vitamin

1/ Supersedes memorandum FI 2017-B.

RESULTS OF EXPERIMENTS ON DOGFISH LIVERS

Composition of Raw Material.	Pretreatment.	Test No.	Processing Method	Oil and Vitamin Recovery from Liver — Oil yield %	Oil recovery %	Vit. A potency Extd oil IU/gram	Vitamin recovery %	Vitamin recovery Million units Per lb. liver
Series 1								
Fresh livers Oil content—73% Oil potency—16,600 IU/gm Liver potency—5.48 million IU per lb.	Fresh livers ground through meat chopper, thoroughly mixed and divided into composite samples.	1	Alkali cook (1%)	65	89	5,900	32	1.77
		2	Water cook	68	93	10,700	60	3.30
		3	Acid cook (½%)	65	89	11,400	61	3.35
Series 2								
Frozen livers Oil content—65% Oil potency—13,400 IU/gm Liver potency—3.95 million IU per lb.	Frozen livers (15 days) ground through meat chopper while frozen, thoroughly mixed and divided into composite samples.	1	Alkali cook (1%)	59	91	12,900	88	3.46
		2	Water cook	53	82	13,600	82	3.20
		3	Acid Cook (½%)	57	88	13,100	86	3.40
Series 3								
Frozen livers Oil content—64% Oil potency—16,100 IU/gm Liver potency—4.67 million IU per lb.	Frozen livers (15 days) ground through meat chopper while frozen, thoroughly mixed and divided into composite samples	1	Alkali cook (1%)	58	91	15,300	87	4.08
		2	Alkali cook (½%)	61	95	14,700	87	4.08
		3	Alkali cook (1%) then ½% acid	54	84	14,900	78	3.64
		4	Alkali cook (1%) then ½% acid	54	84	15,100	79	3.69
		5	Alkali cook (1%) then ¼% acid	61	95	15,100	89	4.15
		6	Water cook	59	92	15,300	87	4.08
Series 4								
Fresh livers Oil content—73% Oil potency—10,500 IU/gm Liver potency—3.50 million IU per lb.	Fresh livers ground through meat chopper, thoroughly mixed and divided into composite samples.	1	Alkali cook (½%)	65	89	4,170	35	1.24
		2	Water cook	60	82	7,330	57	1.99
		3	Acid cook (½%)	62	85	9,620	78	2.73
	One-half of above frozen after grinding and held 5 days.	4	Alkali cook (½%)	64	88	9,050	76	2.65
		5	Water cook	59	81	9,550	74	2.59
		6	Acid cook (½%)	64	88	9,550	80	2.80
Series 5								
Fresh livers Oil content—72% Oil potency—14,100 IU/gm Liver potency—4.62 million IU per lb.	Fresh livers ground through meat chopper, thoroughly mixed and divided into composite samples.	1	Alkali cook (½%)	56	78	4,980	28	1.27
		2	Water cook	58	81	9,690	56	2.56
		3	Acid cook (½%)	65	90	12,700	81	3.74
	Ground livers stored 40°F., 5 days.	4	Cold centrifuging	54	75	6,450	34	1.58
	Cold centrifuge residue—fresh.	4a	Water cook	15.6	22.0	30,800	47	2.18
		4+4a		69.6	97	.....	81	3.76
	Cold certifuge residue—fresh.	4b	Acid cook (¾%) then Alk. 3%	16.3	23.0	26,600	43	1.95
		4+4b		70.3	98	.....	77	3.53
	Cold centrifuge residue—frozen 2 days.	4c	Water cook	15.7	22	33,800	52	2.40
		4+4c		69.7	97	.....	86	3.98
	Cold centrifuge residue—frozen 2 days.	4d	Alkali cook (1%)	15.7	22	31,300	48	2.22
		4+4d		69.7	97	.....	82	3.80
Series 6								
Fresh livers Oil content—66% Oil potency—18,600 IU/gm Liver potency—5.57 million IU per lb.	Fresh livers ground through attrition mill and held 40°F. 2 days.	1	Cold centrifuging	48	73	7,880	31	1.73
	Cold centrifuge residue thoroughly mixed and divided into composite samples.	1a	Acid cook (½%) then 1½% Alk.	15.4	23	32,200	39	2.17
		1+1a		63.4	96	.....	70	3.90
	Cold centrifuge residue—frozen and held 11 days.	1b	Acid cook (1%) then 4% Alk.	13.0	20	37,900	40	2.24
		1+1b		61.0	93	.....	71	3.97
Series 7								
Fresh livers Oil content—72% Oil potency—5,060 IU/gm Liver potency—1.66 million IU per lb.	Fresh livers ground through attrition mill—processed immediately.	1	Cold centrifuging	58	80	1,850	29	0.48
	Cold centrifuge residue—fresh.	1a	Alkali cook (1%)	9.1	12.5	8,060	20	0.33
		1+1a		67.1	92.5	.....	49	0.81
	Cold centrifuge residue—fresh.	1b	Acid cook (1%)	9.7	13.5	15,200	41	0.68
		1+1b		67.7	93.5	.....	70	1.16
	Cold centrifuge residue—frozen 6 days.	1c	Acid cook (1%) then 3% Alk.	11.4	15.8	15,200	47	0.78
		1+1c		69.4	95.8	.....	76	1.26
	Cold centrifuge residue—frozen 6 days.	1d	Water to 212°F. then 1% Alk.	10.5	14.5	13,500	39	0.65
		1+1d		68.5	94.5	.....	68	1.13

extraction. Under these conditions, 80% of the total oil carrying only 29% of the total vitamin was removed by cold extraction.

In the limited work herein reported, the efficiency of recovery of the concentrated vitamin has been considerably below the theoretical maximum. It is believed, however, that further study will lead to refinements making possible an approach to this value. The potentialities of concentration, nevertheless, are readily apparent. In one instance, 52% of the total vitamin was concentrated into and recovered in but 22% of the total oil, which, when considered with the cold extracted oil, made a total of 97% oil and 86% Vitamin A recovery from the livers. The concentrated vitamin oil had a potency of 33,800 IU per gram, as compared to 4,980; 9,690, and 12,700 IU per gram for the total oil removed by alkali, water, and acid cooks, respectively. Fine mechanical disintegration followed by cold extraction permitted subsequent recovery of 47% of the total vitamin of the liver in 15.8% of the oil originally present.

Summary

In summary, the following points appear worthy of mention:

1. In the tests made, high oil recovery from fresh dogfish livers was no guarantee of efficient Vitamin A extraction.

2. Within the limits of the test conditions, Vitamin A was not as readily extracted from fresh livers as it was from frozen livers.

3. Alkali digestion of fresh livers led to poor vitamin recovery, while an acid cook facilitated vitamin extraction.

4. Alkali, plain water and acid cooks gave quite comparable results with frozen livers, effecting efficient extraction of both oil and Vitamin A.

5. When fresh livers were mechanically disintegrated, the greater proportion of the total oil present could be removed by cold centrifuging, leaving the greater proportion of the Vitamin A in the residual liver tissue, thus permitting subsequent extraction of the vitamin in more concentrated form.

6. Because of the significance of these findings with respect to more efficient and flexible utilization of dogfish livers in the production of Vitamin A oil, a further and more detailed study of the problem is now being undertaken.

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